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(12) ABSTRACT OF INVENTION

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(54) METHOD OF MANUFACTURING PERMANENT MAGNETS BASED ON RARE EARTH ELEMENTS AND TRANSITION METAL COMPOUND

(57) Abstract:

FIELD: powder metallurgy. SUBSTANCE: method involves melting of basic and auxiliary compounds; molding workpieces from powder in magnetic field; sintering of workpieces while they are in contact one with the other; separating workpieces from basic compound and using them as magnets. Workpieces from auxiliary compound may be recycled. Magnets obtained from compound Nd<sub>15</sub>Dy<sub>2.5</sub>Fe<sub>15.5</sub>B<sub>7</sub> have variable value of magnetic field intensity through the whole height and changing distribution of

intensity. In case both sides of workpieces are in contact one with the other, uniform distribution of magnetic field intensity through the whole section of magnet (up to 1.5-3.0%) is obtained. In case of gradient distribution, magnetic field intensity may vary from 0 to 3400 Oe, with character of intensity distribution being regulated. EFFECT: wider operational capabilities by providing controllable distribution of magnetic field intensity in magnets and by increasing uniformity of magnetic properties of magnets. 3 cl, 1 tbl

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## (12) SPECIFICATION OF INVENTION TO USSR INVENTOR'S CERTIFICATE

(21), (22) Application: 4352289/02, 29.12.1987 (48) Publication date 20.04.1995 (58) References: Sagawa M. et al. New material for permanent magnets on a base of Nd and Fe – J. of Appl. Phys., v. 55(6), p. 11A, 1984, pp. 2083-2087. European patent EP N 0134305, class H01F 1/04, 1985	(71) Applicant: Filial vsesoyuznogo nii elektromekhan (72) Inventors: Turov V.D., Samukhin G.I., Lobytsev E.S., Rastegaev V.S.
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## (54) METHOD OF MANUFACTURING IRON-BASED PERMANENT MAGNETS

## (57) Abstract

The invention relates to methods of manufacturing iron-based permanent magnets containing neodymium, dysprosium, iron and boron, and may be used in electrical engineering and electronics. An object of the invention is to expand the technological capabilities by creating a controlled distribution of magnetic field strength in the magnets and to increase the homogeneity of magnetic properties of the manufactured magnets. The method involves melting a starting and an auxiliary compounds, pressing compacts from a powder in a magnetic field, and sintering them under conditions of mutual contact. After sintering, the compacts from the starting compound are separated out and used as magnets, while the compacts from the auxiliary compound may be used again. Magnets from a compound  $Nd_{15}Dy_{2.5}Fe_{75.5}B_7$  with a variable value of magnetic field strength along the height and a different distribution of the strength are obtained. In the case of two-sided contacting the compacts, a homogeneity of the magnetic field strength distribution over the cross section of the magnet is achieved to be up to 1.5 – 3.0%. Upon a gradient distribution, the strength may vary from 0 to 3400 Oe, wherein a nature of the strength distribution may be adjusted. 2 dependent claims, 1 table.

### Specification

The invention relates to the powder metallurgy, in particular to methods of manufacturing iron-based permanent magnets containing rare earth metals, mainly neodymium, dysprosium and boron, and may be used in electrical engineering and electronics.

An object of the invention is to expand the technological capabilities by creating a controlled distribution of magnetic field strength in the magnets and to increase the homogeneity of magnetic properties of the manufactured magnets.

The inventive method of manufacturing iron-based permanent magnets comprises melting a starting compound containing neodymium, dysprosium, iron and boron, with an iron content by 0.1-2.0 wt.% higher than the stoichiometric one, and a similar auxiliary compound with an iron content by 2-5 wt.% lower than the stoichiometric one, preparing powders of the starting and auxiliary compounds, pressing compacts therefrom (from the starting compound in a magnetic field), subsequently sintering them under conditions of mutual contact, and heat-treating. If necessary to obtain a homogeneous distribution of the magnetic field strength over the volume of the magnet, a contact of the compacts from the starting compound with the compacts from the auxiliary compound in two opposite planes is provided for during sintering the compacts from the starting compound.

The compacts from the auxiliary compound before contacting with the compacts from the starting compound may be presintered, which simplifies the technology of separating the magnets from the starting compound after sintering due to a reduced welding thereof to the compacts of the auxiliary compound. Furthermore, this makes it possible to use the compacts from the auxiliary compound many times.

The selection of the iron content of the starting and auxiliary iron-based compounds (in the starting compound by 0.1-2.0 wt.% above the stoichiometric content, in the auxiliary compound by 2-5 wt.% below the stoichiometric content) provides for, upon sintering of the compacts under conditions of mutual contact, the realization of an experimentally observed mechanism of diffusion-stimulated disintegration in the compacts with a reduced content of rare earth compounds, with the formation of a R<sub>2</sub>Fe<sub>14</sub>B (R is Nd, Dy) type matrix phase and of grain-boundary interlayers of a soft magnetic phase enriched with iron. The diffusion-stimulated disintegration process does not disturb the texture of the compacts, which is obtained at the step of pressing the powder of the starting compound in the magnetic field.

An increase in the iron content of the starting compound by less than 0.1% above the stoichiometric content results in terminating the diffusion-stimulated disintegration and does not allow a controlled distribution of the magnetic field strength to be obtained in the magnets, while an increase in the iron content by more than 2% above the stoichiometric content slows the diffusion-stimulated disintegration process and is therefore not advisable.

The iron content of the auxiliary compound is also selected so as to provide for an optimum rate of the diffusion-stimulated disintegration process without disturbing the initial texture of the compacts. With an iron content of less than 2 wt.% below the stoichiometric content, the process is terminated, which does allow to achieve the object of the invention, and a reduction in the iron content by more than 5 wt.% as compared with the stoichiometric composition does not result in a further acceleration of the process, but is related to an excessive consumption of the expensive rare earth metal (neodymium, dysprosium).

The inventive method of manufacturing iron-based permanent magnets is implemented by way of example on the manufacture of iron-based permanent magnets from a compound Nd<sub>15</sub>Dy<sub>2.5</sub>Fe<sub>75.5</sub>B<sub>7</sub>. This compound is characterized by the following stoichiometric ratio of the components, wt.-%: Nd 30.5; Dy 5.72; Fe 62.71; and B 1.07. A series of starting compounds and auxiliary compounds with a different component ratio was prepared from the initial components: a neodymium metal NM-1 according to TU-48-4-205-72 (*Note: TU is an abbreviation for the industrial standards in Russia*), a dysprosium metal DYM-1 according to TU-48-4-214-72, a refined carbonyl iron according to TU-12-1-1720-76 and an amorphous boron of B99L grade according to TU-6-02-585-75. The compounds were prepared by melting in an induction furnace

L32-67 in alundum crucibles, followed by pouring into water-cooled steel ingot molds, in an argon atmosphere. The obtained ingots of the compounds were crushed on a jaw crusher down to a dispersity of 200-300  $\mu\text{m}$  and were ground in a planetary mill in an acetone medium down to a dispersity of 2-3  $\mu\text{m}$ . Compacts from the obtained powder of the starting and auxiliary compounds with a diameter  $\varnothing$  of 13 mm and a height  $h$  of 6 mm were pressed at a specific pressure of 4.5 t/cm<sup>2</sup> in magnetic field with a strength of 10-12 kOe, and were then sintered at 1080-1120°C during 0.5-4.0 h and were heat-treated by cooling in accordance with the examples shown below. For comparison, magnets were manufactured from the same compound ( $\text{Nd}_{14}\text{Dy}_{2.5}\text{Fe}_{75.5}\text{B}_7$ ) by a known method.

A change of the magnetic field strength was measured in the manufactured magnets by a Hall sensor along the height of the magnets in the central portion thereof. The measurements were carried out after a preliminary magnetization of the sintered magnets by a pulse magnetic field with a strength of 70 kOe in an axial direction. The measurement of the strength was carried out at 1 mm intervals by using a layer-wise grinding off the magnet from one end.

**Example 1.** Pressed compacts from the starting and auxiliary compounds were assembled in pairs with their opposite poles due to the force of magnetic interaction. The assembled compacts were sintered for 30 min in a vacuum in a SShVL (Sintering Shaft Vacuum Laboratory) type furnace at 1080-1120°C. After sintering, cooling together with the furnace was carried out. The manufactured magnet from the starting compound was separated out from the sintered compact of the auxiliary compound. A magnetization of the manufactured magnet was carried out by a pulse magnetic field with a strength of 70 kOe in an axial direction. Then, a magnetic field strength at the end of the sample in the central portion thereof was measured with a Hall sensor. After the measurement, a 1 mm grinding off the end was made, and a measurement was carried out again, etc. The results are shown in the table.

**Examples 2-5.** The starting and auxiliary compounds with a ratio of the components as indicated in the table were dealt with similarly to the Example 1. A sintering time was varied within 0.5-4.0 h. In Examples 2 and 3, the iron content of the starting and auxiliary compounds corresponds to the invention and extends beyond the invention in Examples 4 and 5.

**Examples 6 and 7.** They were dealt with similarly to Example 1, but the compact from the starting compound was disposed between the compacts from the auxiliary compound. After sintering, the magnet from the starting compound was separated out from the sintered compacts from the auxiliary compound and was inspected.

**Examples 8 and 9.** They were dealt with similarly to Examples 1 and 7, using compacts manufactured by carrying out Examples 1 and 7 as the compacts from the auxiliary compound.

The results of manufacturing magnets from the same compound ( $\text{Nd}_{14}\text{Dy}_{2.5}\text{Fe}_{75.5}\text{B}_7$ ) by the known method using the starting compound of the stoichiometric composition are also shown in the table.

As it follows from Examples 1-3 and 6-9, the inventive method makes it possible to obtain magnets with a controlled distribution of the magnetic field strength and, in Examples 7 and 9, with a homogeneous distribution of the magnetic field strength.

By using the known method, it is not possible to obtain magnets with a controlled distribution of the magnetic field strength similar to that of the magnets manufactured in Examples 1-3, 6 and 8.

In all cases of the manufacture of magnets by the known method, they are characterized by an uncontrolled distribution of the magnetic field strength  $H$  at a heterogeneity coefficient  $K_H$

$$\left( K_H = \frac{H_{\max} - H_{\min}}{H_{\max}} \right)$$

of up to 20%.

When similar magnets are manufactured with a homogeneous distribution of the magnetic field strength according to the present invention (Examples 7 and 9), the heterogeneity coefficient decreases down to 1.5-3.0%.

The magnets manufactured by the inventive method are also characterized by a higher level of magnetic characteristics, which is evidenced by an increase in the general level of magnetic field strength. A possibility for repeated use of the sintered compacts from the auxiliary compound as contacting compacts when carried out the inventive method follows from Examples 8 and 9.

Use of the inventive method makes it possible to expand the technological capabilities during the manufacture of iron-based permanent magnets and to increase the effectiveness of their application in different fields of modern engineering.

**Claims:**

1. A METHOD OF MANUFACTURING IRON-BASED PERMANENT MAGNETS, comprising melting a starting compound containing neodymium, dysprosium, iron and boron, preparing a powder, pressing compacts in a magnetic field, sintering and heat-treating the compacts, characterized in that, in order to expand technological capabilities by creating a controlled distribution of magnetic field strength in the magnet, the starting compound is melted with an iron content that is by 0.1-2.0 wt.% higher than the stoichiometric content, an auxiliary compound is additionally melted with an iron content that is by 2-5 wt.% lower than the stoichiometric content, a powder is prepared and compacts are pressed from the auxiliary compound, and a contacting of the pressed compacts from the starting and auxiliary compounds is provided during the sintering.
2. The method according to claim 1, characterized in that, in order to increase a homogeneity of magnetic properties of the manufactured magnets, the contacting of the compacts from the starting and auxiliary compounds is carried out along two opposite surfaces of the compact from the starting compound.
3. The method according to claims 1 and 2, characterized in that, before the contacting, the pressed compacts from the auxiliary compound are presintered.

Example	Magnet manufacture method	Ratio of components in compounds, wt.%							
		starting compound				auxiliary compound			
		Nd	Dy	Fe	B	Nd	Dy	Fe	B
	Known	30.5	5.72	62.71	1.07	—	—	—	—
1	Invention	30.42	5.70	62.81	1.07	32.13	6.09	60.71	1.07
2	Same	29.69	5.53	63.71	1.07	32.94	6.28	59.71	1.07
3	Same	28.87	5.35	64.71	1.07	34.56	6.66	57.71	1.07
4	Beyond the invention	30.47	5.70	62.76	1.07	31.72	6.00	61.21	1.07
5	Same	28.06	5.16	65.71	1.07	35.38	6.84	56.71	1.07
6	Invention	30.42	5.70	62.81	1.07	34.56	6.66	57.71	1.07
7	Same	30.42	5.70	62.81	1.07	32.13	6.09	60.71	1.07
8	Same	30.42	5.70	62.81	1.07	32.13	6.09	60.71	1.07
9	Same	29.69	5.53	63.71	1.07	32.94	6.28	59.71	1.07

Continuation of table

Example	Position of compacts from starting and auxiliary compounds during sintering	Sintering time	Magnetic field strength of (H, Oe) at a distance from the end of magnet, mm					
			0	1	2	3	4	5
	Starting compound	1-4	2700	2200	2300	2600	2300	2400
1		0.5	3000	1000	500	0	0	0
2		2.0	3200	2500	2000	1500	500	0
3	Auxiliary compound	4.0	3400	3000	2600	2000	1000	300
4	Starting compound	0.5	50	0	0	0	0	0
5		4.0	2800	50	0	0	0	0
6	Auxiliary compound	0.5	3000	1000	500	500	1000	3000
	Starting compound							
7	Auxiliary compound	3.0	3300	3250	3300	3300	3250	3300
8	As in Example 1	0.5	2900	1200	300	100	0	0
9	As in Example 7	3.0	3300	3250	3200	3300	3300	3300